

CARBON DIOXIDE BINDING IN THE HETEROGENEOUS SYSTEMS FORMED AT COMBUSTION OF OIL SHALE 4. REACTIVITY OF ASHES TOWARDS ACID GASES IN THE SYSTEM FLY ASH–FLUE GASES

T. KALJUVEE*, R. KUUSIK, M. RADIN

Tallinn Technical University,
Department of Basic and Applied Chemistry
5 Ehitajate Rd., 19086 Tallinn, Estonia

V. BENDER

Tallinn Technical University,
Department of Physics
5 Ehitajate Rd., 19086 Tallinn, Estonia

The results obtained studying the binding efficiency of different ashes formed at Estonian Power Plant (EPP) and in the fluidized-bed combustion facilities towards CO₂ and SO₂, and the possibilities of activation of these ashes are presented. Experiments were carried out with thermogravimetric equipment at isothermal conditions in the temperature range of 400–800 °C in the atmosphere of CO₂–air (+/–H₂O) or SO₂–air mixture. Chemical, X-ray diffraction, IR spectroscopy, BET nitrogen dynamic desorption, and other methods were used for characterizing the initial samples and the products of experiments.

The CO₂- as well as SO₂-binding efficiency of ash samples taken from EPP was quite poor without preliminary activation. The best binding occurred at 750–800 °C. After a 5-min contact between solid and gaseous phases (at CO₂ or SO₂ partial pressure 190 mmHg) 0.06–0.12 mmole CO₂ or 0.10–0.15 mmole SO₂ was bound per 100 mg sample depending on the kind of ash. Preliminary activation of the samples increased their binding capacity towards CO₂ and SO₂ up to 2.2 and 3.8 times, respectively, reaching the level of binding efficiency of ashes formed in fluidized-bed conditions. The results of these studies confirm that the reactivity of oil shale ashes at binding acid gases greatly depends on ash formation conditions as well as on the binding conditions and preliminary activation of ashes.

* Corresponding author: e-mail tiidu@edu.ttu.ee

Introduction

The use of fossil fuels in the energy production causes serious pollution of the atmosphere with different toxic and environmentally unfriendly compounds as SO_2 , CO , CO_2 , NO_x , etc. Electricity production in the Republic of Estonia is based mainly on pulverized firing (at flame temperature 1200–1400 °C) of Estonian oil shale (OS) [1, 2] which is characterized by low content of organic (~30%) and high content of mineral matter, from which approximately 50% are carbonates (calcite, dolostone).

The carbonates decompose during OS combustion with the formation of CO_2 , and free Ca and Mg oxides, which are the main binders of acid gaseous compounds. Due to the high residual content of Ca and Mg oxides in the ashes they should have a considerable reactivity towards acid gaseous compounds being potential sorbents for deeper purification of flue gases. It was well proved by industrial-scale experiments at Baltic Power Plant (PP) for desulphurisation of flue gases by recurrent use of cyclone ash [3, 4]. An idea to utilize oil-shale ash for fixation of CO_2 into solid phase is discussed in [5], and the amounts of fixed CO_2 are calculated on the basis of theoretical considerations and experimental studies [6–8].

Mitigation of CO_2 emissions is of great importance and numerous methods have been proposed to capture CO_2 from flue gases. The methods base on chemical and physical absorption, membrane technology and chryogenic processes [9–11]. According to the existing data, 73–91% of CO_2 can be bound, the expenses needed for that being on the level of 400–1200 EEK per t of CO_2 [12, 13].

Physical adsorption of CO_2 for its recovery from flue gases at coal (fossil fuels) combustion was studied using a molecular sieve [14] or pelletized Ca-X-type adsorbent [15] both based on zeolite. At pilot-plant testing the 90-% removal of 99% pure CO_2 was achieved [14]. Another idea for recovery of CO_2 on combustion of fossil fuels – coal [16] and natural gas [17] – is based on partial recirculation of flue gases in order to maximize CO_2 concentration in them. A method based on adsorption of CO_2 by lime in fluidized-bed adsorber-desorber is of great interest [18].

Our previous paper [6] presented the results of studying the activity of different oil shale ashes formed at Estonian and Baltic PPs towards CO_2 binding in model and field conditions at moderated temperatures – from –5 to 20 °C as well as the theoretical and practical limits of this approach [7, 8]. Considering the absence of investigations dealing with the activity of OS ashes towards CO_2 binding at higher temperatures, the aim of the present work was the comparative study of the CO_2 - and SO_2 -binding efficiency of different ashes formed at Estonian PP as well as in the fluidized-bed combustion facilities at a temperature range of 400–800 °C, including the influence of preliminary activation of these ashes.

Experimental

Materials

Five samples of different ashes and, for comparison, a limestone sample from *Karinu* deposit and a dolostone sample from *Hellamaa* deposit were investigated. Two ash samples – bottom ash (HBA) and cyclone ash (HCA) were formed at 1200–1400 °C (high-temperature ashes, HTA) at combustion of Estonian oil shale at Estonian PP. The other three ashes were formed at 800–950 °C (low-temperature ashes, LTA), one at combustion of lumpy OS in the 1-MWth CFBC (Ahlström Pyroflow test facility, Karhula, Finland), and others at combustion of lumpy semicoke (SC) in the laboratory BFBC (Tallinn Technical University). The ashes were activated by grinding (G) or by grinding with subsequent hydrating of samples (G + H) [19]. The main characteristics of the samples studied are presented in the Table.

The high-temperature ashes slightly differ from each other in the content of CaO_{total} and CaO_{free} , insoluble residue, etc. (see the Table). The grinding and especially grinding with subsequent hydrating increased specific surface area (SSA) value of HTA. The grinding of oil shale ash from CFBC increased both CaO_{free} content and SSA of the sample. Semicoke ashes formed in laboratory BFBC contain less CaO_{total} and CaO_{free} , but more insoluble residue than oil shale ash from CFBC. *Karinu* limestone and *Hellamaa* dolostone contain 52.6 and 28.8% CaO_{total} , 1.5 and 25.5% MgO and 1.1 and 0.5% insoluble residue, respectively. Their SSA was 3.0 and 1.2 m²/g.

Methods

The experiments were carried out with thermogravimetric equipment (Q-derivatograph, MOM and *Labsys*, Setaram), the samples were calcinated in dynamic heating conditions (5 or 10 K per min) in the atmosphere of air, and carbonised or sulphated in isothermal conditions (at the temperature interval 400–800 °C) in the atmosphere of CO₂–air or SO₂–air mixture.

The calcination was performed by heating the sample to 800–850 °C in order to achieve its constant weight when necessary; the sample was cooled down to the required temperature, and thereafter the air enriched with CO₂ or SO₂ was led into the reaction chamber. Multiplate Pt-crucibles were used, thickness of the sample layer 0.2–0.3 mm, sample mass ~100 mg, gas flow rate 270 ml per min and partial pressure of CO₂ or SO₂ in the gas mixture 144 or 190 mmHg. The influence of humidity on the binding efficiency of CO₂ was studied at the CO₂ partial pressure 144 mmHg adding water vapor by bubbling the gas stream through pure water before leading it into the reaction chamber at the temperature guaranteeing the relative humidity of gases on the level of 30%. In *Labsys* equipment standard 100- μ L Pt-crucibles were used, weight of samples ~20 mg, air flow rate 40 mL per min.

Main Characteristics of the Samples

Parameter	Sample						
	Ashes					Karuru limestone	Hellamaa dolostone
	HBA	HCA	LBA (BFBC)		LBA (CFBC)		
			800 °C	950 °C			
Content, wt.%, of:							
CaO _{total}	54.9	49.1	42.8	39.8	45.0	52.6	28.8
CaO _{free} :							
in initial samples	20.8	13.1	–	–	23.8	–	–
in activated samples (G)	22.7	18.7	25.4	23.7	30.0	–	–
MgO	5.7	5.1	–	–	9.4	1.5	25.5
SO ₄ ²⁻	4.5	6.9	9.6	8.9	18.4	0	0.5
Al ₂ O ₃ + Fe ₂ O ₃	10.3	12.6	–	–	6.5	0.3	0.6
SiO ₂	21.1	23.5	24.7	26.7	12.9	1.1	0.5
BET-surface area, m ² /g:							
of initial samples	0.76	0.62	–	–	1.91	2.98	1.24
of activated samples:							
ground (G)	1.45	1.01	3.82	2.40	3.87	–	–
ground and hydrated (G+H)	4.84	3.33	–	–	–	–	–
Content, wt.%, of the fractional class $-45\mu\text{m}$:							
in initial samples	23.7	58.5	–	–	3.1	99.5	100
in activated samples (G)	89.3	89.8	99.3	98.9	92.3	–	–

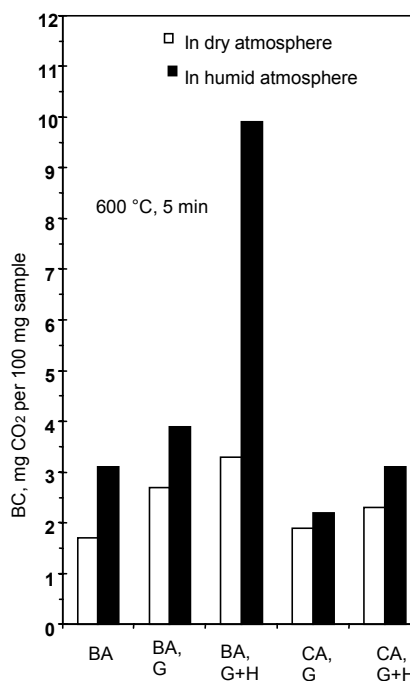
Three parameters were used to characterize the extent of interaction: the CO₂(SO₂)-binding capacity (mg or mmole of CO₂ or SO₂ per 100 mg sample), CO₂(SO₂)-binding rate (mmole or mg CO₂ or SO₂ per mg sample · min), and CO₂(SO₂)-binding efficiency, %, per content of CaO_{free} (or for natural carbonate samples per content of total CaO and MgO).

Different methods of analysis (chemical, BET nitrogen dynamic desorption, X-ray, IR-spectroscopy, etc.) were used for characterization of initial samples and of treatment products.

Results and Discussion

The CO₂(as well as SO₂)-binding efficiency of HBA without preliminary activation was quite low – during a 5-min contact between gaseous and solid phases in the atmosphere containing 15% CO₂ at 600 °C 1.7 mg CO₂, and at 750 °C (the optimum temperature for CO₂ binding by ashes in dry atmosphere) 5.5 mg CO₂ were bound per 100 mg of sample. The prolongation of the contact time up to 30 min at 600 and 750 °C increased the binding capacity (BC) of HBA up to 2.4 and 7.7 mg CO₂ per 100 mg of sample, respectively. Preliminary grinding increased BC of the samples to the 40-% level. For instance, at 600 °C during a 5-min contact HBA bound 2.7 mg CO₂ per 100 mg of sample.

Fig. 1. CO₂-binding capacity (BC) of bottom (HBA) and cyclone (HCA) ash samples (G – ground; G + H – ground and hydrated) in dry gaseous (15% CO₂) and in humid (+ H₂O) atmosphere



At the same conditions the ground sample of HCA, due its lower content of CaO_{free}, bound 1.9 mg CO₂ per 100 mg of sample. Combined activation of HBA and HCA samples by grinding with subsequent hydration increased the amount of bound CO₂ up to 3.3 and 2.3 mg per 100 mg of sample, respectively (Fig. 1). The CO₂-binding rate of the ground HCA and HBA samples at the first seconds of the contact was in the range of 0.017–0.020 mg CO₂ per mg of sample · min and that for the ground + hydrated samples – 0.021 and 0.032 mg CO₂ per mg of sample · min, respectively.

Humidity of gaseous phase influenced the reactivity of ashes towards CO₂ considerably at lower temperatures – 400–600 °C, especially when testing previously ground and hydrated samples. In these experimental conditions (600 °C and a 5-min contact) HCA and HBA bound 3.1 and 9.9 mg CO₂ per 100 mg of sample, respectively, or 1.3 and 3 times more than in dry gaseous atmosphere (see Fig. 1). The CO₂-binding rate of HCA and HBA in the first seconds of the contact between gaseous and solid phases was 0.03 and 0.10 mg CO₂ per mg of sample · min, respectively, or 1.4 and 3 times higher than in dry gaseous atmosphere as well.

The results obtained confirmed that in dry gaseous atmosphere in the temperature interval from 400 to 750 °C, the CO₂-binding capacity of the samples increased, especially at 600–750 °C. So, in dry gaseous atmosphere at 750 °C during a 5-min contact the ground and hydrated HBA bound 11.2 mg CO₂ per 100 mg of sample, or 4 times more compared with that at 600 °C (Fig. 2).

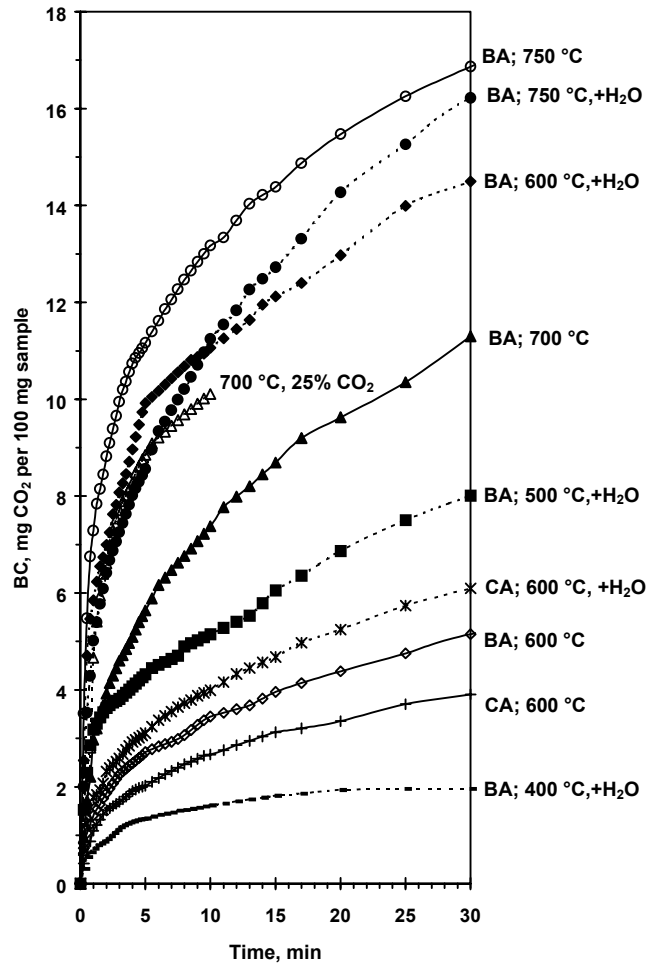


Fig. 2. Dependence of CO_2 -binding capacity (BC) of preliminary ground and hydrated bottom (HBA) and cyclone (HCA) ash samples on temperature in dry gaseous (15% CO_2) and in humid (+ H_2O) atmosphere

In the first seconds of the contact the CO_2 -binding rate was 0.03 and 0.15 mg CO_2 per mg of sample \cdot min at 600 and 750 $^\circ\text{C}$, respectively, and during a 30-min contact 20.7 and 68.2% of CaO_{free} present in the samples was utilized (Figs 3 and 4).

The supporting influence of humidity on binding characteristics of the samples is well illustrated by the fact that the CO_2 -binding capacity and binding rate of activated HBA as well as the degree of CaO_{free} utilized at 600 $^\circ\text{C}$ in the atmosphere containing water vapor are comparable with the results obtained at 750 $^\circ\text{C}$ in dry atmosphere (see Fig. 2). At the temperatures higher than 750 $^\circ\text{C}$ the decomposition of carbonates started to prevail.

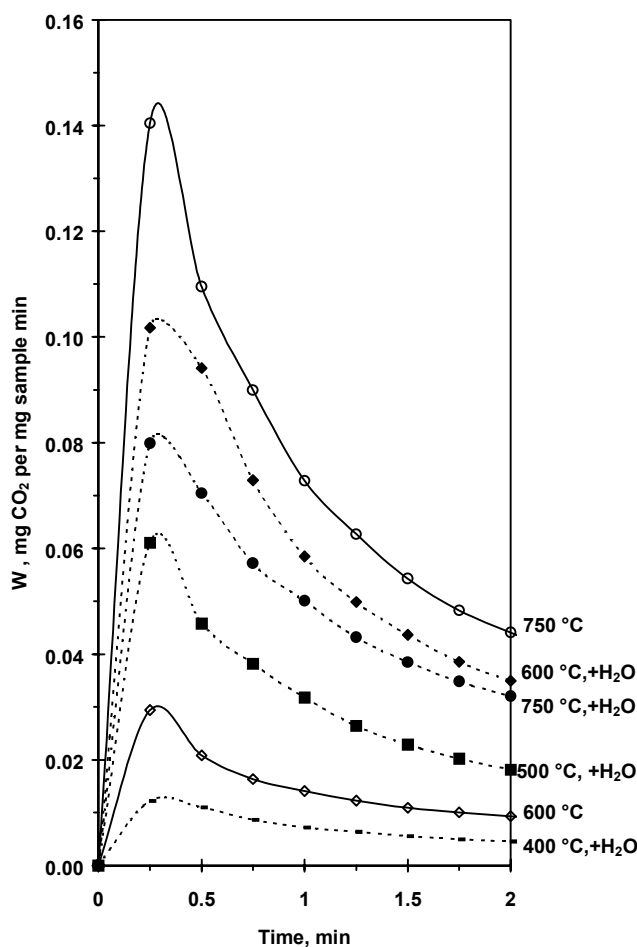


Fig. 3. Dependence of CO_2 -binding rate (W) of preliminary ground and hydrated bottom ash sample on temperature in dry gaseous (15% CO_2) and in humid (+ H_2O) atmosphere

The differences in the CO_2 -binding capacities and CO_2 -binding rates at different temperatures in dry gaseous atmosphere were on average 30–35% higher for HBA compared with the values for HCA. The differences in the humid atmosphere were especially considerable – 2-3 times (see Fig. 2) depending on the experiment temperature.

The influence of CO_2 partial pressure on the binding characteristics of ashes was determined at 700 °C. Increasing the CO_2 partial pressure in dry gaseous atmosphere from 144 to 190 mmHg at testing ground samples of HCA and HBA, the BC of samples increased on average 10 and 20% (rel.), and at testing ground and hydrated samples – 20 and 50% (rel.), respectively.

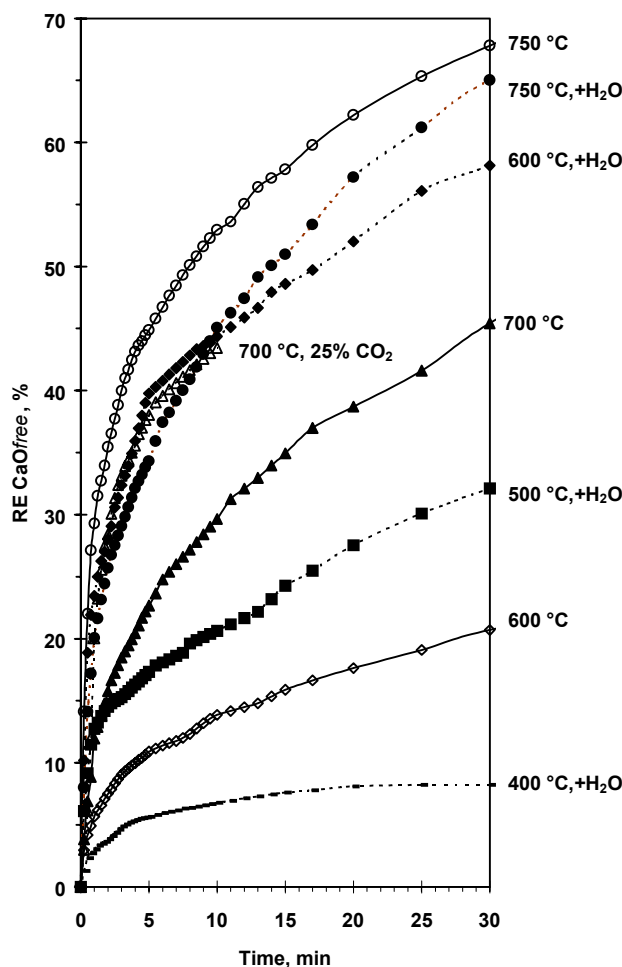


Fig. 4. Dependence of CO₂-removal efficiency per CaO_{free} content of preliminary ground and hydrated bottom ash sample (RE CaO_{free}) on temperature in dry gaseous (15% CO₂) and in humid (+ H₂O) atmosphere

For example, the CO₂-binding capacity of ground HBA increased during a 5-min contact from 5.2 to 6.7 mg, and that of ground and hydrated HBA from 5.6 to 8.9 mg per 100 mg of sample. During a 10-min contact 29.7 and 43.4% of CaO_{free} present in the above samples were utilized, respectively (see Figs 2–4).

An interesting effect in TG, DTG and DTA curves was observed comparing the thermograms of Estonian OS and its mixtures with different coal samples with and without oil shale ash addition obtained at thermooxidation of these blends. Oil shale and coal mixture (mass ratio 1 : 1) with addition of oil-shale ash at the mole ratio of CaO_{free}/S 1.7 and 3.0 gave an endoeffect in DTA curves with minimums at 746 and 750 °C, respectively, instead of 744 °C fixed for the sample without ash addition.

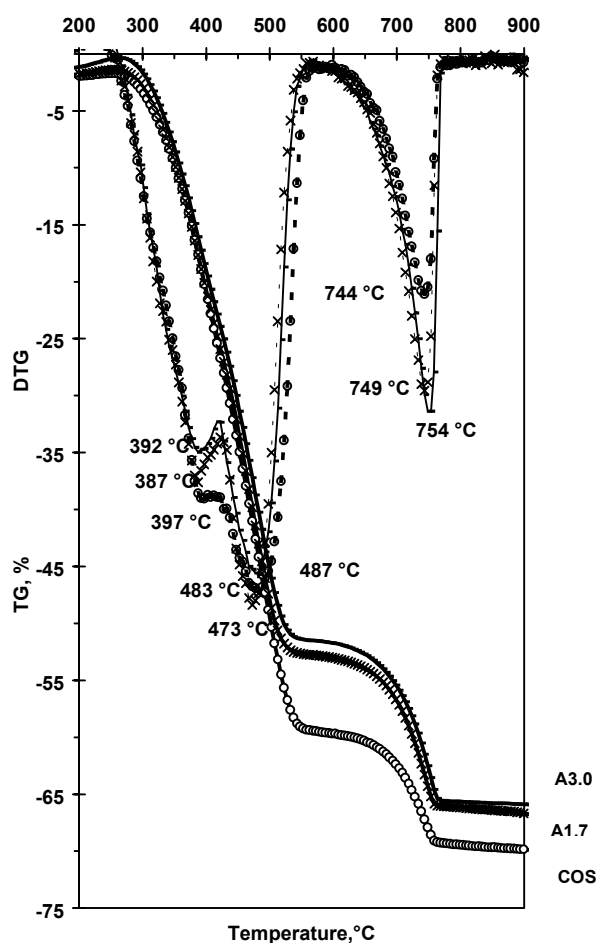


Fig. 5. TG and DTG curves for coal and oil-shale mixture (COS; mass ratio 1 : 1) with and without oil shale ash (A) addition at mole ratio of $\text{CaO}_{\text{free}}/\text{S}$ 1.7 and 3.0

The mass loss minimums in DTG curves were fixed at 749 and 754 °C (instead of 744 °C), respectively. These changes correspond to the decomposition of secondary carbonates formed from CaO_{free} in oil-shale ash during thermooxidation of fuel organic matter at temperatures below 550–570 °C.

The additional mass loss (3.5 and 4.5%, respectively) followed the decomposition of secondary carbonates in oil shale–coal mixture with ash addition at the same mole ratios (Fig. 5). This fact also indicates that at using fluidized-bed technique, a part of CO_2 formed at combustion of solid fossil fuels can be bound into the solid phase (ash) in the following gas-tract at moderated temperatures (<550–600 °C).

CO_2 - and SO_2 -binding characteristics of ground ash samples were determined in dry gaseous atmosphere containing CO_2 or SO_2 keeping their

partial pressure on the level of 190 mmHg. The SC ashes formed in the laboratory BFBC at 950 and 800 °C bound during a 2-min contact 0.16 and 0.23 mmole CO₂ per 100 mg sample, respectively, or 1.5 and 2.3 times more than bound by HBA in the same experimental conditions. LBA formed in the BFBC at 800 °C bound during a 2-min contact 0.28 mmole CO₂ per 100 mg sample, or 2.8 times more than HBA. The CO₂-binding capacity of ashes formed at 800–850 °C (both in BFBC and in CFBC) was 1.5–1.9 times higher than that of the ash formed at 950 °C in BFBC.

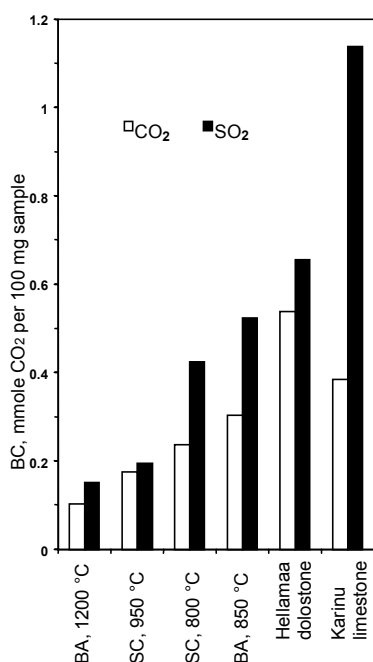


Fig. 6. CO₂- and SO₂-binding capacity (BC) of different samples: oil-shale bottom ash (BA) of Estonian PP (formed at 1200 °C); semicoke (SC) formed in laboratory BFBC at 800 or 950 °C; oil-shale bottom ash (BA) of Karhula CFBC (formed at 850 °C); Karinu limestone and Hellamaa dolostone preliminary calcined at temperature up to 900 °C – all treated under the same conditions: 700 °C, 2-min contact, $p\text{CO}_2(\text{SO}_2) = 190 \text{ mmHg}$

Calcinated samples of Karinu limestone and Hellamaa dolostone bound 0.38 and 0.53 mmole CO₂ per 100 mg sample, respectively, or 3.8 and 5.3 times more than HBA and 1.4–1.7 and 1.9–2.3 times more than the ashes formed at 800–850 °C (Fig. 6).

In analogous experimental conditions in the atmosphere containing SO₂, HBA bound during a 2-min contact 0.15 mmole SO₂ (1.5 times more than CO₂) per 100 mg sample. The ashes formed in BFBC at 950 and 800 °C bound 0.18 and 0.41 mmole SO₂, respectively, or 1.5 and 2.7 times more than they bound CO₂ (2.2 and 2.7 times more SO₂ than bound by HBA), and LBA formed in CFBC bound 0.53 mmole SO₂ (2.1 times more than CO₂), i.e. 3.5 times more SO₂ than bound by HBA, per 100 mg sample. So, the SO₂-binding capacity of ashes formed at 800–850 °C was 2.2–2.9 times higher than that of the ashes formed at 950 °C. Karinu limestone and Hellamaa dolostone bound 1.14 and 0.6 mmole SO₂ per 100 mg sample or 7.6 and 4.4 times more, respectively, than HBA, and 2.2–2.8 and 1.2–1.6 times more than the ashes formed at 800–850 °C (Fig. 6).

So, the results obtained confirmed that the conditions of ash formation, especially temperature and aerodynamics, influence their physical and chemical properties (SSA, content of CaO_{free} , etc.) considerably, and that their activity towards binding acid gaseous compounds. The influence of SSA on the CO_2 - and SO_2 -binding ability of the samples studied is well demonstrated in Fig. 7.

According to the better formation conditions, the LTA have better binding characteristics towards CO_2 and SO_2 than HTA, and the fluidized-bed, especially CFBC technique, is an advanced one compared with pulverized-firing technique for solid fossil fuels. However, considering that HTA formed at oil shale pulverized firing at power plants have a considerably high residual reactivity towards acid gaseous compounds (CaO_{free} content 15–25%), they could be considered potential sorbents for deeper cleaning of flue gases by dry method. For recycling of these ashes the right injection point (optimum temperature and aerodynamical conditions for CO_2 and SO_2 binding) is to be chosen and activated ashes are to be used [3]. For determination of actual binding parameters at actual partial pressures of CO_2 and water vapor as well as at conditions of simultaneous content of CO_2 and SO_2 in flue gases, additional measurements should be carried out.

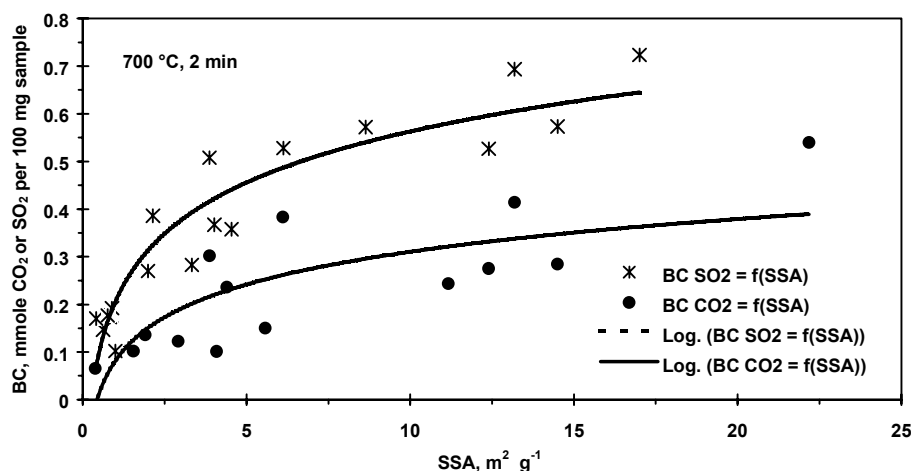


Fig. 7. Dependence of CO_2 - and SO_2 -binding capacity (BC) of different lime-containing materials on the sample SSA

The results of X-ray analysis of initial ash samples, carbonation and sulphation products as well as of preliminary calcinated limestone and dolostone samples confirmed that only CaO , not MgO took part in binding of CO_2 , as the only product of carbonisation of the samples studied was CaCO_3 . Both MgO and CaO took part in binding SO_2 , and in the 700°C ashes, besides of CaSO_4 , $\gamma\text{-CaSO}_4$ and $\text{CaMg}_3(\text{SO}_4)_4$, traces of CaS were identified.

Conclusions

The investigations carried out showed that the reactivity of OS and SC ashes in binding acid gaseous compounds greatly depends on ash formation conditions as well as on the binding conditions and on the preliminary activation of ashes.

The binding ability of initial HTA towards CO₂ (as well as towards SO₂) was comparatively low, but the preliminary activation of ashes increased their activity up to 2 times. Increasing the temperature from 500 to 700–750 °C increased the CO₂-binding characteristics of activated HTA approximately 2 times. The humidity of the gaseous atmosphere increased the CO₂-binding capacity of ashes at the lower temperatures (400–600 °C) additionally up to 2.5 times. As calcium hydroxide decomposes at higher temperatures (>600 °C), the additional CO₂-binding effect was not observed. Ground LTA bound approximately 2 times more CO₂ than ground HTA and 1.4 times more than ground and hydrated HTA.

Comparison of the CO₂- and SO₂-binding ability of the samples studied showed that HBA bound 1.5 times more SO₂ than CO₂, and LTA, depending on the temperature of binding and their formation conditions, 2.0–2.7 times more SO₂ than CO₂. Karinu limestone and Hellamaa dolostone bound 3.0 and 1.2 times more SO₂ than CO₂, respectively. These differences are caused, firstly, by the very low reactivity of MgO towards CO₂ and, secondly, by the CaCO₃ shell formation on the surface of the particles that hinders the diffusion of CO₂ inside the particles more than CaSO₄–CaMg₃(SO₄)₄ shell hinders the diffusion of SO₂. Thermodynamical considerations also give the preference to SO₂ at interactions investigated [7].

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